United States Patent

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[72] Inventor **Arthur B. Broerman**
Bartlesville, Okla.

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[73] Assignee **Phillips Petroleum Company**

[54] **CHROMATOGRAPHIC ANALYZER SAMPLE VALVE**
6 Claims, 5 Drawing Figs.

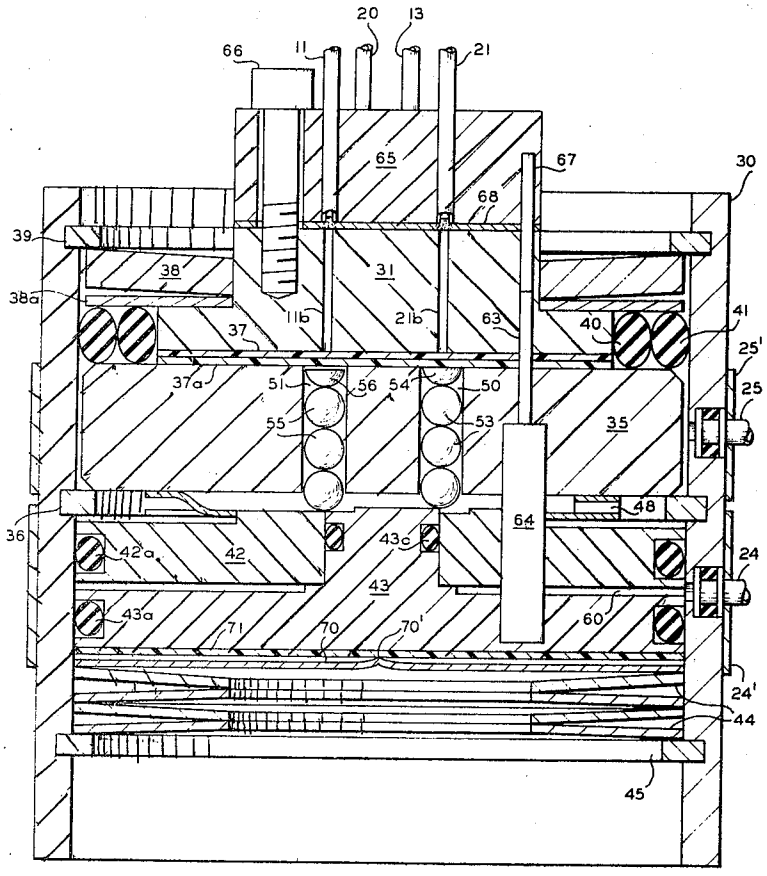
[52] **U.S. Cl.** ..... **73/422 GC, 55/197**
[51] **Int. Cl.** **G01n 1/00**
[50] **Field of Search** ..... 73/422 GC; 251/62

[56] **References Cited**
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2,955,618	10/1960	Miller	251/62
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Primary Examiner—S. Clement Swisher
Attorney—Young and Quigg

ABSTRACT: A sample valve for a chromatographic analyzer is actuated by a pair of pistons which move plungers to block communication between selected valve ports. The valve components are contained within a cylindrical housing by split rings and spring washers. The force exerted by the washers on one end of the valve assembly is concentrated by a cylindrical plate having a central protrusion.



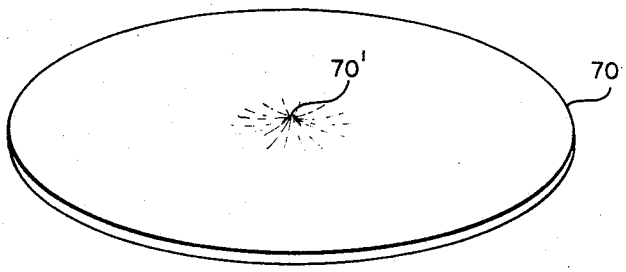


FIG. 5

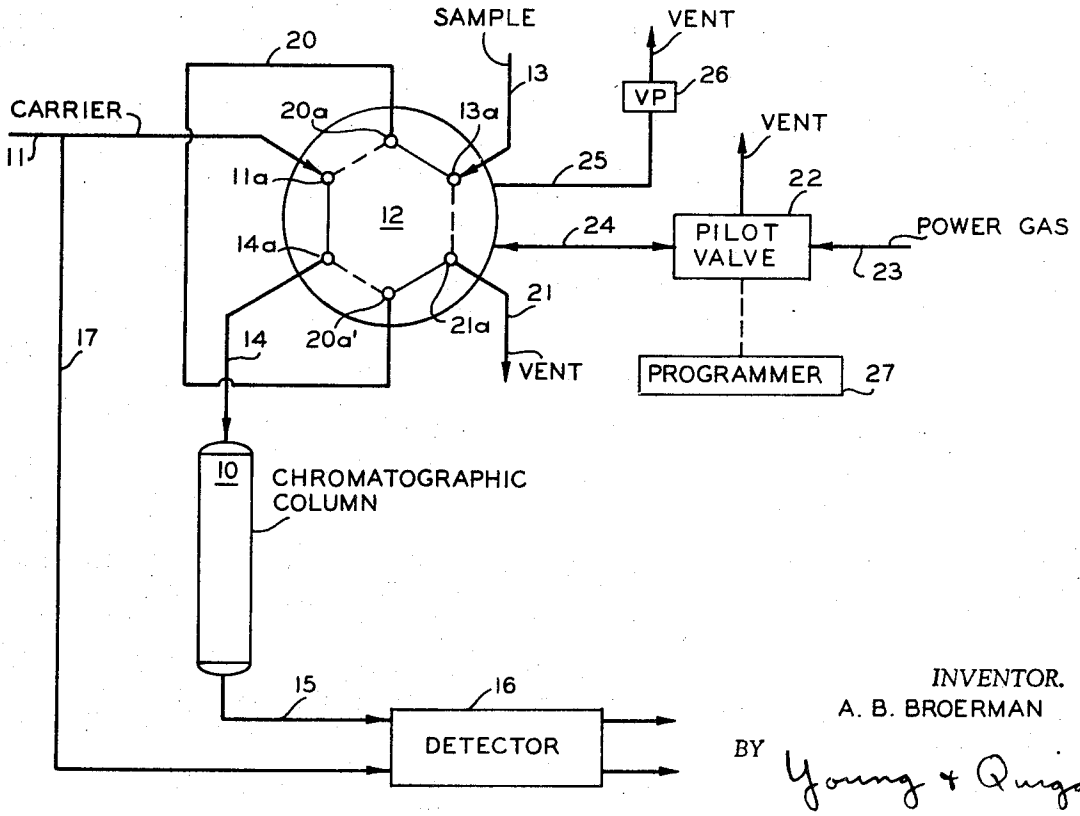


FIG. 1

INVENTOR.
A. B. BROERMAN

BY Young & Quigg

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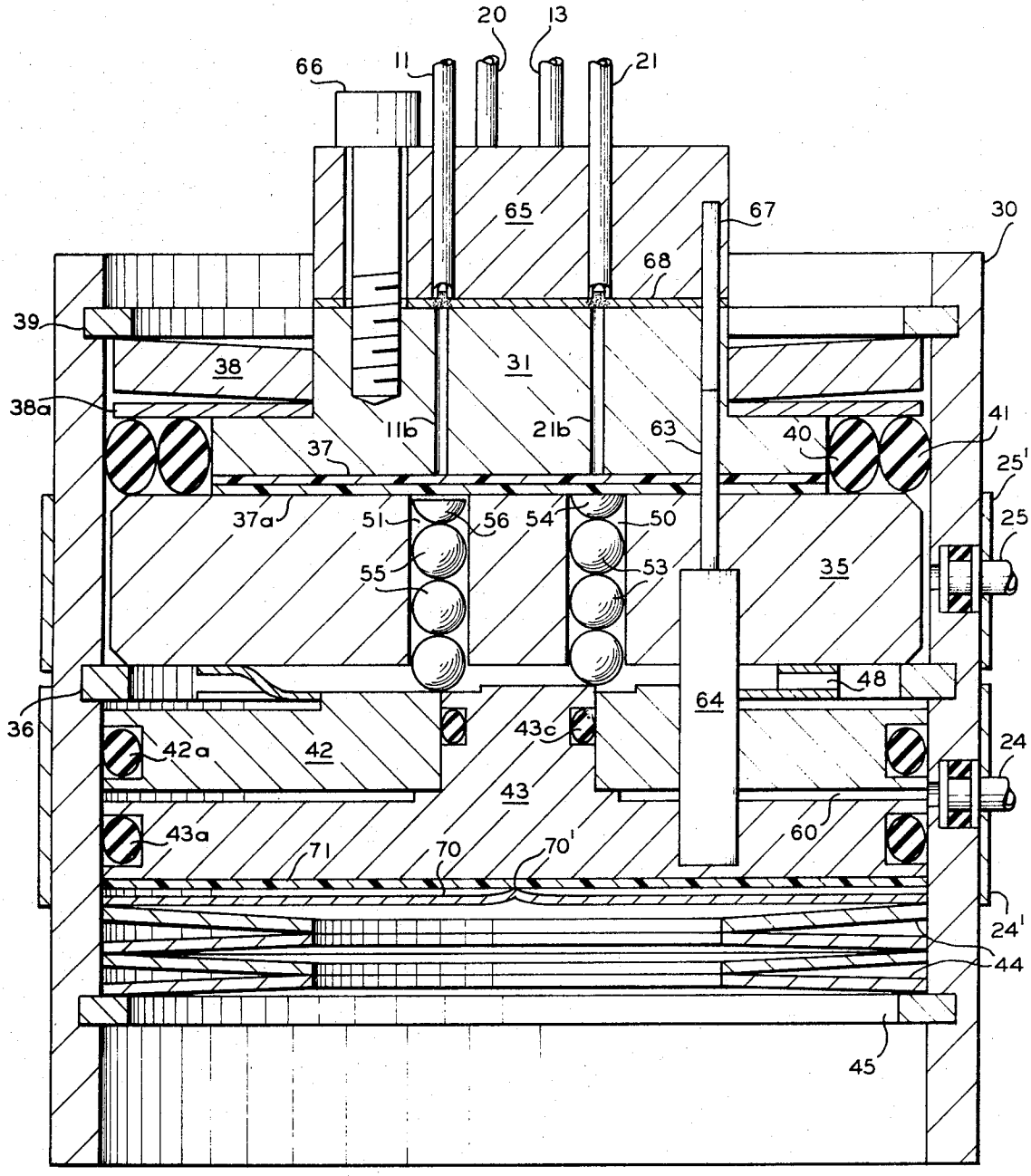


FIG. 2

INVENTOR.
A. B. BROERMAN

BY Young & Quigg

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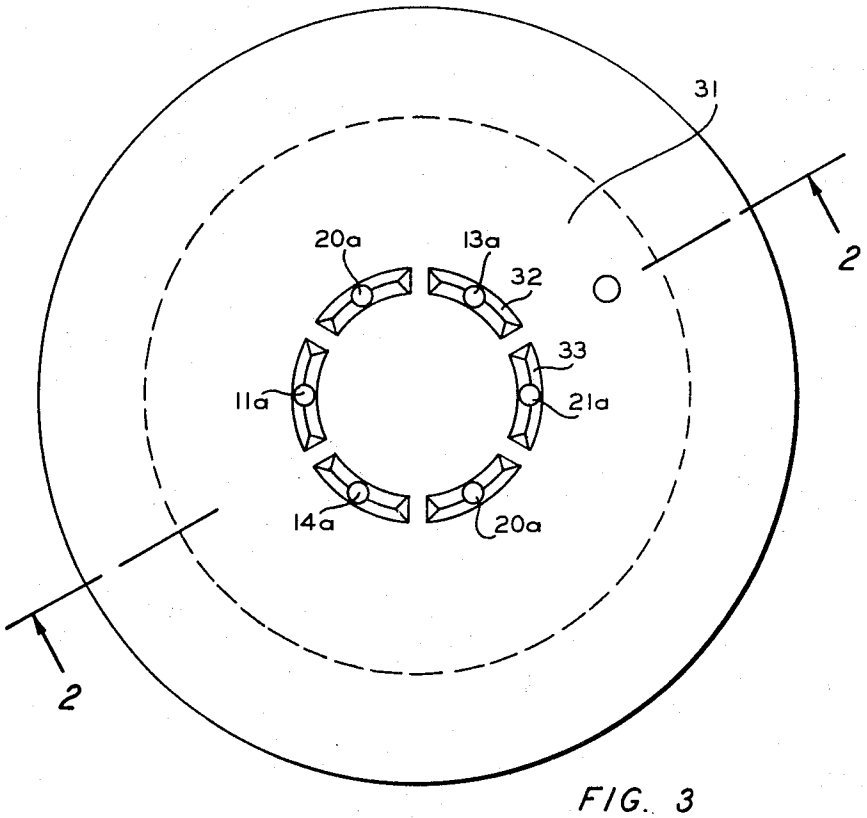


FIG. 3

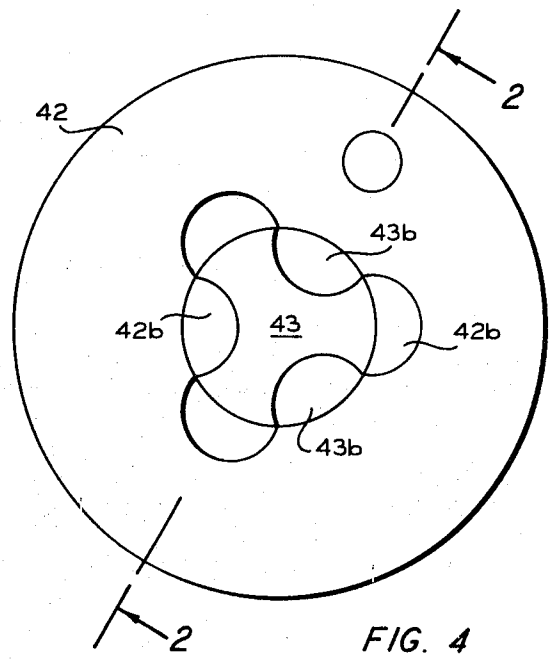


FIG. 4

INVENTOR.
A. B. BROERMAN

BY Young & Quigg

ATTORNEYS

CHROMATOGRAPHIC ANALYZER SAMPLE VALVE

It is common practice to analyze fluid mixtures by means of chromatography. In a conventional chromatographic analyzer, a sample of the material to be analyzed is introduced into a chromatographic column, and carrier gas is thereafter passed through the column to elute the constituents of the sample in sequence. It is important that the sample volumes remain constant in order to obtain reproducable results for a series of analyses. This can be accomplished by means of sample valves which trap a predetermined volume of the sample in a loop and deliver this trapped volume to the column when the valve is actuated. A number of pneumatically operated diaphragm valves have been designed which are particularly effective for this purpose.

One such valve is described in my copending application, Ser. No. 796,718, filed Feb. 5, 1969 now U.S. Pat. No. 3,545,491. The components of the valve described therein are contained within a cylindrical housing. Split rings are positioned in grooves within the housing to retain the valve components in predetermined positions. Spring washers are employed to bear against the split rings and exert forces on the valve components. Movable pistons are contained within the housing to displace plungers which selectively block passages between spaced ports of the valve. These plungers are arranged in a circular path with alternate plungers being moved together in response to the valve being actuated. The resulting assembly is analogous to a three-legged stool which can be somewhat unstable under certain conditions.

In accordance with the present invention, a valve of the foregoing type is improved by inserting a circular plate between the springs and one of the pistons which actuates the plungers. The plate is provided with a center protrusion which concentrates the force of the springs at essentially a single point. This provides a more stable valve assembly and permits the valve to handle fluids at higher pressures.

In the drawing,

FIG. 1 is a schematic representation of a chromatographic analyzer having the sample valve of this invention incorporated therein.

FIG. 2 is a view, shown partially in section, of the sample valve employed in the analyzer of FIG. 1.

FIG. 3 illustrates the lower face of the cap of the valve of FIG. 2.

FIG. 4 is a top view of the movable piston in the valve of FIG. 2.

FIG. 5 is a perspective view of the force concentrating plate employed in the valve of this invention. Referring now to the drawing in detail and to FIG. 1 in particular, there is shown a chromatographic column **10** which contains any suitable packing or partitioning material. Carrier fluid is introduced through a conduit **11** which communicates with a first inlet port **11***a* of the sample valve **12**. This carrier fluid can either be a gas or a liquid, depending on the particular packing material and the desired separation. A sample fluid to be analyzed is introduced through a conduit **13** which communicates with an inlet port **13***a* of valve **12**. A conduit **14** extends from a valve port **14***a* to the inlet of column **10**. A conduit **15** extends from the outlet of column **10** to the first inlet of a detector **16**. A portion of the carrier fluid is directed through a conduit **17** to a second inlet of detector **16**. As is well known in the art, detector **16** can be any suitable instrument which is capable of providing an output signal that is representative of differences in composition of the two fluids passed thereto.

Sample valve **12** is employed to introduce a predetermined volume of sample selectively to the inlet of column **10**. This is accomplished by means of a sample loop **20** which extends between ports **20***a* and **20***a'*. The volume of sample loop **20** is selected in accordance with the desired volume of sample to be introduced into the valve. When the valve is in the position illustrated, the ports are connected in the directions shown by the solid lines. Carrier fluid thus flows from conduit **11** to conduit **14**. Sample material flows from conduit **13** through sample loop **20** and is vented through a conduit **21** which communicates with a port **21***a*. When the sample valve is actuated, the ports are connected in the directions shown by the illustrated broken lines. At this time, carrier fluid displaces the volume of sample trapped in sample loop **20** and forces this sample into column **10**. The incoming sample is passed to vent conduit **21** at this time. Sample valve **12** is pneumatically operated and receives operating pressure from a pilot valve **22**. Power gas is introduced into valve **22** through an inlet conduit **23**. A conduit **24** extends between the pilot valve and sample valve **12**. A conduit **25** extends from valve **22** to a vacuum pump **26** to facilitate operation of the sample valve under certain conditions, as described hereafter in greater detail. The operation of the sample valve can be controlled by a programmer **27** which actuates pilot valve **22** at predetermined intervals. Operation of the sample valve by pilot valve **22** is described in greater detail in U.S. Pat. No. 3,376,894.

Sample valve **12** is illustrated in FIGS. **2, 3** and **4**. The valve components are contained within a cylindrical housing **30**. These components include a valve cap **31** which is connected to conduits **11, 20, 13, 21** and **14** of FIG. **1**. These conduits communicate with respective passages, two of which are shown as passages **11***b* and **21***b*, that extend from the upper surface of cap **31** to spaced points on the lower surface which lie in a circular path. The lower ends of these passages constitute the ports illustrated in FIG. **3**. As shown therein, the lower surface of valve body **31** is provided with a plurality of recesses which extend part of the distance between adjacent ports. For example, a recess **32** extends from port **13***a* toward port **21***a*, and a recess **33** extends from port **21***a* toward port **13***a*. Corresponding recesses are provided adjacent each of the ports in the lower surfaces of valve cap **31**.

A cylindrical valve body **35** is positioned within housing **30** and supported on a split-retaining ring **36** which is snapped into a central recess in the housing. Two diaphragms **37** and **37***a* are disposed between cap **31** and body **35**. The upper diaphragm can advantageously be formed of polymerized tetrafluoroethylene, and the lower diaphragm **37***a* can advantageously be formed of polyester. However, a single diaphragm can be employed if desired. A spring, such as a Belleville washer **38**, surrounds the upper portion of cap **31** and engages a second split-retaining ring **39** which is inserted in an upper recess in housing **30**. A washer **38***a* is positioned between washer **38** and cap **31**. Annular sealing rings **40** and **41** are disposed in the region between body **35**, cap **31** and washer **38***a*.

Two movable pistons **42** and **43** are positioned within housing **30** beneath body **35**. These pistons are supported by a stack of Belleville washers **44** which rests on a third split-retaining ring **45**. A cylindrical plate **70**, which has a central protrusion **70'**, is positioned between the top washer **44** and a dust-sealing disk **72** which engages the lower surface of piston **43**. Disk **71** can be formed of a plastic material such as polymerized tetrafluoroethylene. Plate **70** is illustrated in FIG. **4**. As will be explained hereafter in greater detail, the purpose of plate **70** is to concentrate the force of the washers at the central region of piston **43**. A spring **48** is positioned between body **35** and upper piston **42** to exert a downward force on this piston. Piston **42** carries a sealing ring **42***a*, and piston **43** carries sealing rings **43***a* and **43***c*.

Body **35** is provided with six vertical passages, two of which are shown as passages **50** and **51**. The upper ends of these passages lie in the circle defined by the six ports in the lower face of cap **31**, with the upper end of each passage being located between a pair of adjacent ports in the cap. For example, the upper end of passage **50** lies between ports **21***a* and **13***a*. Movable plungers are disposed in each of the six passages in body **35**. As illustrated, the plunger in passage **50** comprises three stacked spheres **53** having a half-sphere **54** at the top of the stack. Similar elements are disposed in the other five passages which extend through body member **35**. Spheres **55** and a half-sphere **56** are shown in passage **51**. If desired, these spherical elements can be replaced by cylindrical rods.

Pistons 42 and 43 are designed to control movement of the plungers through the passages of body 35. As illustrated in FIGS. 2 and 3, the elevated upper surface of piston 43 engages the stack of spheres in passage 50 so as to move half-sphere 54 into engagement with the lower surface of diaphragm 37*a*. This serves to compress diaphragms 37 and 37*a* between ports 13*a* and 21*a* so as to block communication between these ports. The lowermost sphere 55 in passage 51 rests on a cutout portion of piston 43 and on piston 42 so that half-sphere 56 in passage 51 is depressed from diaphragm 37*a* when the valve is in the position illustrated. The plungers in the remaining passages in body 35 are connected in a corresponding manner with alternate plungers engaged diaphragm 37*a* when the valve is in the position illustrated.

Conduit 24 of FIG. 1 communicates with a chamber 60 between pistons 42 and 43. Conduit 25 communicates with a region above piston 42. Conduits 24 and 25 are advantageously secured to housing 30 by respective spring-retaining rings 24' and 25' which partially surround the housing and are provided with slots to engage the ends of the conduits. Pins 63 and 64 are provided to maintain alignment between elements 31, 35, 42 and 43 when the valve is assembled.

In the absence of power gas flowing into chamber 60 of FIG. 2, the valve is in the position illustrated. Washers 44 and plate 50 exert an upward force on piston 43, the upper surface of which engages lowermost sphere 53 so that the region of diaphragms 37 and 37*a* engaged by half-sphere 54 is moved upwardly to block communication between ports 13*a* and 21*a*. Communication is also blocked between ports 11*a*, 20*a* and 14*a*, 21*a* by corresponding plunger means, not shown. This serves to connect the ports in the directions illustrated by the solid lines in FIG. 1. The pressures of the flowing fluids serve to depress diaphragms 37 and 37*a* into those recesses illustrated in FIG. 3 which are not blocked by the elevated plungers.

When pilot valve 22 is switched to the second position, power gas is introduced into chamber 60 from conduit 24. As the pressure builds up in chamber 60, a force is exerted to move piston 42 upwardly against the force of retaining spring 48. This results in the upper surface of piston 42 engaging lowermost spheres 55 to raise spheres 55 and 56 through passages 51. As the pressure continues to increase in chamber 60, sufficient force is built up to overcome the force of washers 44 so that piston 43 is moved downwardly. This lowers spheres 53 and 54. The combined movement of the two groups of spheres thus switches the valves to the position illustrated by the dotted lines in FIG. 1.

In a second embodiment of the valve operation, vacuum pump 26 of FIG. 1 is connected by conduit 25 to the region above piston 42. This permits operation with carrier gas and/or sample fluid of substantially lower pressures because the application of vacuum to the underside of diaphragm 37*a* through passages 50 and 51 assist in the downward displacement of the diaphragm. In this embodiment it is often desirable to connect the pilot valve vent conduit to the inlet of vacuum pump 26. This removes residual atmospheric pressure from chamber 60 when an air signal is not being applied. Otherwise, there is a danger of this residual pressure overcoming spring 48.

It is often advantageous to connect the inlet conduits to the valve of this invention by means of a manifold 65, as shown in FIG. 2. Manifold 65 is provided with a plurality of spaced passages to receive the inlet conduits. These passages terminate adjacent the corresponding passages in cap 31. Manifold 65 can be attached to cap 31 by a plurality of screws 66. Correct alignment between members 31 and 65 is assured by means of a pin 67. A sealing gasket 68 is disposed between members 65 and 31. In a preferred embodiment, this gasket is formed of a compressible porous material such as sintered silver. When screws 66 are tightened, the porous material is compressed to form a seal. However, the six spaced regions at the ends of the inlet conduits are not so compressed, with the result that the original porous material forms a filter to remove any particulate material that might be present in the carrier gas or sample material.

As previously mentioned, an important feature of this invention resides in the use of circular plate 70 which is provided with central protrusion 70'. This serves to concentrate the force of Belleville washers 44 at the center of piston 43. The concentration of force at this region prevents any rocking motion which might otherwise occur due to the fact that the upper portion of the piston engages three spaced plungers. It has been found that this construction permits operation of the valve with samples at substantially higher pressures than could be employed heretofore.

While this invention has been described in conjunction with a presently preferred embodiment, it should be evident that it is not limited thereto.

What is claimed is:

1. In a sample valve which includes a housing enclosing a body having six longitudinal passages therethrough which terminate in a generally circular first path at a first face of the body, a cap having six longitudinal passages therethrough which terminate in a generally circular second path at a first face of the cap, said first and second paths having substantially the same diameter, a diaphragm positioned adjacent the first face of said body, means urging said cap against said diaphragm so that the first face of said cap is adjacent the first face of said body member and said first and second circular paths are adjacent one another with the longitudinal passages in said cap terminating between the longitudinal passages in said body member, a first annular piston positioned adjacent said body on the side thereof remote from said cap, a second piston positioned adjacent said first piston, said second piston having a central section which extends through said first piston, said first and second pistons being of such configuration that a chamber is formed therebetween, said housing having an opening therein which communicates with said chamber, spring means positioned to exert a force on said second piston urging said second piston toward said first piston, and plungers positioned in each of the passages in said body member, the lengths of said plungers and the configurations of said pistons being such that first alternate plungers engage said diaphragm in the absence of external fluid pressure being applied to said chamber and second alternate plunger means engage said diaphragm when external fluid pressure is applied to said chamber; the improvement comprising a plate having a central protrusion thereon, said plate being positioned between said spring means and said second piston so that said protrusion engages said second piston and concentrates the force of said spring means at a central region of said second piston relative to said first circular path.

2. The sample valve of claim 1 further comprising a disk positioned between said second piston and said plate and engaging the walls of said housing to form a seal therewith.

3. The sample valve of claim 1 wherein said spring means comprises a plurality of Belleville washers.

4. The sample valve of claim 1 further comprising a manifold secured to said cap, said manifold having six passages therethrough which terminate in communication with respective passages through said cap.

5. The sample valve of claim 4, further comprising a gasket formed of compressible, normally porous material positioned between said cap and said manifold so that the gasket is compressed to become nonporous when the manifold is secured to the body, with regions of said gasket adjacent said passages remaining noncompressed and porous.

6. The sample valve of claim 5 wherein said gasket is formed of sintered silver.

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